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Microstructural Evolution in Austenitic Stainless Steels Irradiated by Neutrons with Improved Control

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Nucleation and growth of interstitial type dislocation loops were investigated by fusion neutron irradiation and fission neutron irradiations with improved control. The density of loops formed by both irradiations increased linearly with the fluence. The average size of loops also increased. The rate equation analysis showed that the loops were nucleated directly by the defect processes in a cascade zone, not by the reaction among the freely migrating point defects in the matrix. If the rate of loop formation is assumed to be directly proportional to the energy of a primary knock-on atom (PKA) produced by a neutron, the threshold energy of 80 keV and the rate of loop formation of 0.05 were obtained.

KEYWORDS: austenitic stainless steel, neutron irradiation, loop formation, PKA energy

1. Introduction

To develop the fusion reactor materials from the results of fission reactor irradiation, it is important to understand the process of microstructural evolution in materials and it is necessary to establish the fission-fusion correlation. Recent development of irradiation techniques in the Japan Materials Testing Reactor (JMTR) at the Oarai Research Establishment of Japan Atomic Energy Research Institute, such as irradiation using multi-section removable irradiation rig to choose the neutron fluence at our demand without the exposure to neutrons at temperatures different from that designed, enabled us to obtain reliable data for these studies.

Understanding of the nucleation of defect clusters is quite important for the defect structure evolution. The nucleation of stacking fault tetrahedra in fcc metals has been studied well and concluded that stacking fault tetrahedra are formed in a cascade zone directly. Each stacking fault tetrahedron represents each subcascade in a cascade at low temperatures[1]. Resent experimental results also show that interstitial type dislocation loops are considered to be formed in a cascade zone, and grow larger by absorption of freely migrating point defects[2].

In this paper first we will show that the nucleation rate of interstitial type dislocation loops is quite low when we assume that the nucleation occurs only by the clustering of freely migrating point defects which escaped from cascade zone. Secondly we will estimate the threshold value of PKA energy for the nucleation of interstitial type dislocation loops by comparison of the results between fission and fusion neutron irradiations.

2. Experimental procedure

In the fission neutron irradiation, Fe-16Cr-17Ni alloy was irradiated in JMTR at 473 K to fluences in the range from 3.6×10^{21} to 2.5×10^{23} n/m^2 (E>1MeV). Here, temperature- and fluence-controlled irradiation rig was used, and it allowed the removal of some specimens out of the reactor irradiation region during full-power reactor operation. Whereas in the fusion neutron irradiation, Fe-15Cr-16Ni alloy was irradiated with D-T neutrons in the Rotating Target Neutron Source-II (RTNS-II) at LLNL at 473 K to fluences of 1.9 to 5.4×10^{22}n/m^2. Each specimen was polished for electron microscopy observation after irradiation, and observed with JEOL TEM 200CX and 2000FX operating at 200keV.

3. Result

3.1 Comparison of defect structures between fission and fusion neutron irradiations

Dark-field weak-beam images of microstructures formed by fission and fusion neutron irradiations are shown in Figs. 1 and 2. Dislocation loops were observed in all cases examined. Dose

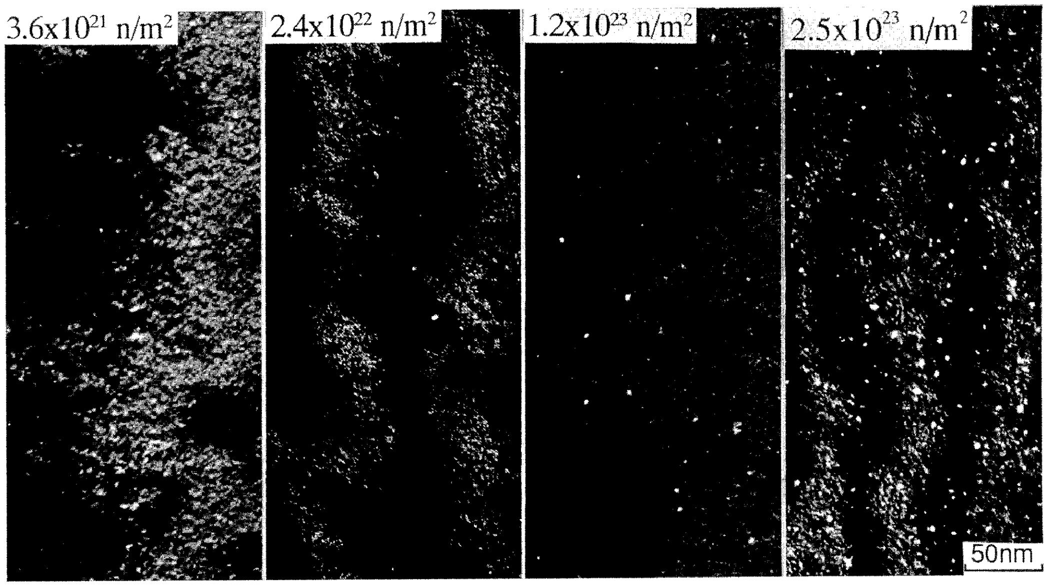

Fig. 1 Dark-field weak-beam images of microstructures formed in Fe-16Cr-17Ni by fission neutron irradiation with JMTR.

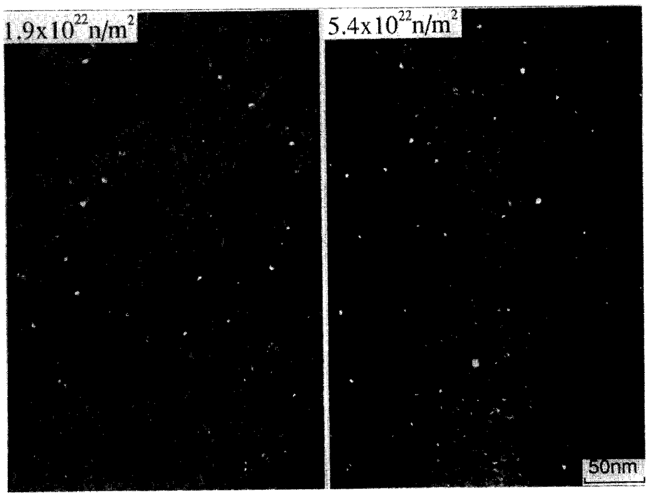

Fig. 2 Dark-field weak-beam images of microstructures formed in Fe-15Cr-16Ni by fusion neutron irradiation with RTNS-II.

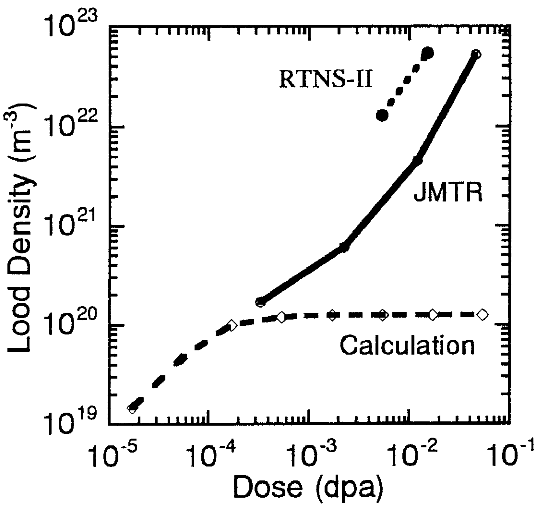

Fig. 3 Dose dependence of loop density in Fe-Cr-Ni alloys.

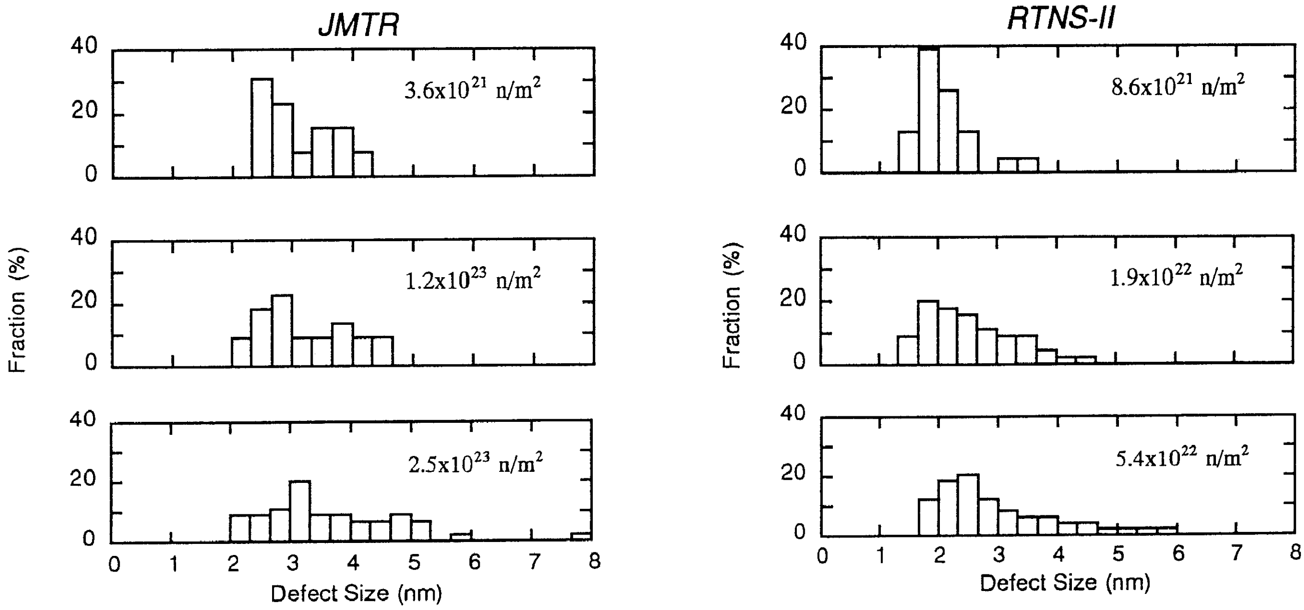

Fig. 4 Size Distribution of loops formed by fission (JMTR) and fusion (RTNS-II) neutron irradiations.

dependence of loop density obtained from Figs. 1 and 2 is shown in Fig. 3. The loop density increased almost linearly with increasing irradiation dose. The size distribution of loops is shown in Fig. 4. The loop size also increased with irradiation dose.

3.2 The rate equation analysis

In order to investigate the nucleation of interstitial type dislocation loops, the analysis by the rate equations was employed, where displacement of atoms is assumed to occur in the form of Frenkel pairs and defect production is homogeneous in space and in time. In the present calculations, only a simplified case is considered. The features of that are as follows:

(1) Mobile defects are single interstitials and single vacancies.

(2) Di-interstitials are stable enough to be the nuclei of interstitial clusters.

(3) The effect of impurities is ignored.

The rate equations, in fractional units, are given by

$$dC_I/dt=P(1-C_V)(1-Z_{IV}C_V)-Z_{IV}(M_I+M_V)C_IC_V -2Z_{II}M_IC_I^2-Z_{LI}M_I(C_{LI}C_L)^{1/2}C_I-Z_{SI}M_IC_{SI}C_I \quad (1)$$

$$dC_V/dt=P(1-C_V)(1-Z_{IV}C_V)-Z_{IV}(M_I+M_V)C_IC_V -Z_{LV}M_V(C_{LI}C_L)^{1/2}C_V-Z_{SV}M_VC_{SV}C_V \quad (2)$$

$$dC_L/dt=Z_{II}M_IC_I^2 \quad (3)$$

$$dC_{LI}/dt=Z_{LI}M_I(C_{LI}C_L)^{1/2}C_I-Z_{LV}M_V(C_{LI}C_L)^{1/2}C_V \quad (4)$$

Meanings of the symbols used in the equations are listed below:

P: generation rate of a Frenkel pairs

C_I: interstitial concentration

C_V: vacancy concentration

C_L: interstitial loop concentration

C_{LI}: concentration of interstitials which have already been absorbed to the loops of concentration C_L

The saturated concentration of interstitial type dislocation loops is given by:

$$C_{LS}=\beta P^{1/2}\exp(E_m^I/2kT) \quad (5)$$

where E_m^I is the migration energy of interstitial, and β the material dependent constant. In the present calculation, E_m^I, P and β are estimated experimentally to be 0.9 eV, 3.7x10^{-8}dpa/s and 1.5x10^{-9}, respectively.

The result of numerical calculation using the rate equations is shown in Fig. 3. The loop density almost increased linearly with the fluence at beginning of irradiation, and then saturated. The experimental results were, however, much higher than the calculated result. These results suggest that the interstitial type dislocation loops are nucleated directly by the defect processes in a cascade zone, not by the reaction among freely migrating point defects in the matrix.

3.3 PKA energy analysis

If a threshold value of PKA energy to form a loop directly in a cascade exits, and the rate of loop formation is proportional to the PKA energy produced by a neutron, the formation of loop can be written as

$$Y_{Loop}=\alpha N_0 \phi t \int_{E_{d(Loop)}}^{E_{p,max}} \frac{E_p}{2E_{d(Loop)}} \frac{d\sigma_p(E_p)}{dE_p} dE_p \quad (6)$$

where α, N_0, ϕ, t and $E_{d(Loop)}$ are the efficiency of loop formation, atomic density, flux, irradiation time and threshold energy of loop formation, respectively. $d\sigma_p(E_p)/dE_p$ is the differential cross-section of primary recoil energy spectrum. Similar equation was used for the analysis of subcascade structure in Cu[3].

The threshold energy, which satisfies simultaneously the results of fission and fusion neutron irradiations, is 80 keV and the rate of loop formation is 0.05. For the calculation, the recoil energy spectra obtained by Shimomura[4] were used. The threshold energy of 80keV is much higher than expected one. In self ion irradiatied Ni and other metals, the formation of loops has been observed by irradiation lower than 80keV. The discrepancy is considered as follows. 1) The high energy part of primary recoil energy spectrum is mainly produced by the nuclear reaction (n, 2n), however, the loop formation by (n, 2n) reaction is unclear. 2) In the present study, it is supposed that the rate of loop formation is directly proportional to PKA energy produced by neutrons. The rate of loop formation may be high at high PKA energy.

For the understanding of the nucleation of point defect clusters by high energy neutron irradiation, it is necessary to clarify the effects of the cascade damage. This work is one trial to understand the nucleation of interstitial type dislocation loops. Further relevant data and calculations are required to obtain more reliable values.

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